

Chemical and Biological Protection and Detection in Fabrics for Protective Clothing

Heidi L. Schreuder-Gibson, Quoc Truong,
John E. Walker, Jeffery R. Owens,
Joseph D. Wander, and Wayne E. Jones Jr.

Abstract

Military, firefighter, law enforcement, and medical personnel require high-level protection when dealing with chemical and biological threats in many environments ranging from combat to urban, agricultural, and industrial. Current protective clothing is based on full barrier protection, such as hazardous materials (HAZMAT) suits, or permeable adsorptive protective overgarments, such as those used by the U.S. military. New protective garment systems are envisioned that contain novel features, such as the capability to selectively block toxic chemicals, to chemically destroy toxic materials that contact the fabric, and to detect hazardous agents on the surface of the fabric. New technologies being built into advanced fabrics for enhanced chemical and biological protection include selectively permeable membranes, reactive nanoparticles, reactive nanofibers, biocidal fabric treatments, and conductive-polymer indicators on optical fibers.

Keywords: *advanced fabrics, antimicrobial treatments, biological protection, chemical detection, chemical protection, permeable membranes.*

Introduction

Chemical and biological (CB) protective clothing is available with many different fabric materials for specific applications. Hazardous materials (HAZMAT) response suits are made from impermeable film-laminated fabrics, fully sealing the wearer from the passage of air, vapors, and liquids. One such suit is the military's toxicological agents protective (TAP) suit, designed to be worn in rugged terrain that may be contaminated with persistent toxic chemicals. It must have a surface that is easily decontaminated with liquids for a minimum of

five reuses. The suit is made with Chemfab's Challenge Ultra Pro fabric system. This is a multilayer laminated fabric system that consists of five alternating layers of fluoropolymer [nonporous poly(tetrafluoroethylene) films] and Nomex nonwoven aramid fiber and a conductive layer. The TAP suit is specialty equipment, designed to work with a self-contained breathing apparatus. In contrast, standard-issue military chemical protection is provided through liquid repellency in conjunction with vapor and air exchange in order to

maintain comfort over longer operational periods at higher workloads. This is accomplished through the use of a semipermeable adsorptive carbon liner within the clothing. The carbon layer within the fabric provides protection by adsorbing vapors that pass into the clothing.

For the military, a suite of chemical protective suits is available for the soldier, each garment choice meeting different protection levels, depending upon the demands of the mission. For example, lightweight, flexible garments have been the mainstay of troop protection for the U.S. military. The battledress over-garment (BDO) and the recently introduced joint service lightweight integrated suit technology (JSLIST), shown in Figure 1, are air-permeable carbon-based overgarments that are designed to be worn over battledress duty uniforms in all environments where personnel are under an immediate threat of a chemical or biological attack.

New Technologies for Protective Fabrics

CB protective fabrics essentially function as barriers to chemical contaminants such as organic liquids and vapors. The fabrics can be constructed to be impermeable, permeable, semipermeable, or selectively permeable. Many HAZMAT suits are made of barrier-fabric laminates that are completely impermeable, such as Tyvek-reinforced polypropylene film laminates with a rugged outer-shell fabric such as aluminized Kevlar and polybenzobisoxazole (PBO), worn with butyl rubber gloves. Permeable protective fabrics contain an activated-carbon layer, such as carbon-loaded open-cell foam or bonded carbon spheres (Figure 2), to adsorb toxic vapors. Semipermeable fabrics incorporate new membrane structures that diminish airflow through the fabric layers, but allow high water-vapor permeability to reduce heat stress for the wearer.

Recently, membranes have been developed for fabrics that allow the permeation of water-vapor molecules, but not larger organic molecules. These membranes are called "perm-selective" membranes, due to the selectivity they exhibit with respect to molecular solubility and diffusion through the polymer structure. Polymer membranes that exhibit a high level of water-vapor permeability, but are resistant to the permeation of organic molecules provide protection from hazardous organic chemicals while allowing a mechanism for water-vapor transport and evaporative cooling through the fabric layer for comfort, as shown schematically in Figure 3.

The U.S. Army has developed new selectively permeable membranes (SPMs)

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14. ABSTRACT Military, firefighter, law enforcement, and medical personnel require high-level protection when dealing with chemical and biological threats in many environments ranging from combat to urban, agricultural, and industrial. Current protective clothing is based on full barrier protection, such as hazardous materials (HAZMAT) suits, or permeable adsorptive protective overgarments, such as those used by the U.S. military. New protective garment systems are envisioned that contain novel features, such as the capability to selectively block toxic chemicals, to chemically destroy toxic materials that contact the fabric, and to detect hazardous agents on the surface of the fabric. New technologies being built into advanced fabrics for enhanced chemical and biological protection include selectively permeable membranes, reactive nanoparticles, reactive nanofibers, biocidal fabric treatments, and conductive-polymer indicators on optical fibers.					
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Figure 1. The joint service lightweight integrated suit technology (JSLIST), an air-permeable carbon-based chemical protective overgarment, with a water-repellent-treated cotton/nylon ripstop fabric shell, a carbon-bonded tricot liner, face mask, butyl rubber gloves, and poly(vinyl chloride) overboots; designed to be worn over battledress duty uniforms in all environments where military personnel are under an immediate threat of a chemical or biological attack.

with membrane specialists such as W.L. Gore & Associates Inc., Acordis, and W.R. Grace & Co. These SPMs allow selective diffusion of water molecules for evaporative cooling while providing protection against CB agents with minimum heat stress, weight, and bulkiness for the wearer.¹ SPM technology developments have their origins in membranes developed for gas separation, water purification by reverse osmosis, and medical applications.^{2,3} Materials for SPMs have been made of films of poly(vinyl alcohol), cellulose acetate, cellulosic cotton, and poly(allylamine). These SPMs have been tested and are effective barriers to CB agents.

Chemical warfare agents penetrate the skin rapidly and can be lethal (skin permeability rates for mustard and nerve agents are 2.0 cm/min and 0.1 cm/min, respectively). While biological warfare agents do not typically penetrate the skin,

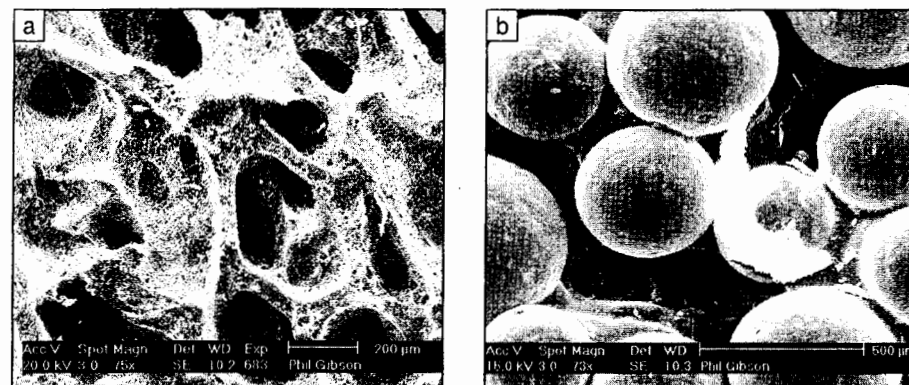


Figure 2. Scanning electron micrographs of (a) activated-carbon-loaded open-cell foam and (b) bonded activated-carbon spheres, used in permeable protective fabrics to adsorb toxic vapors.

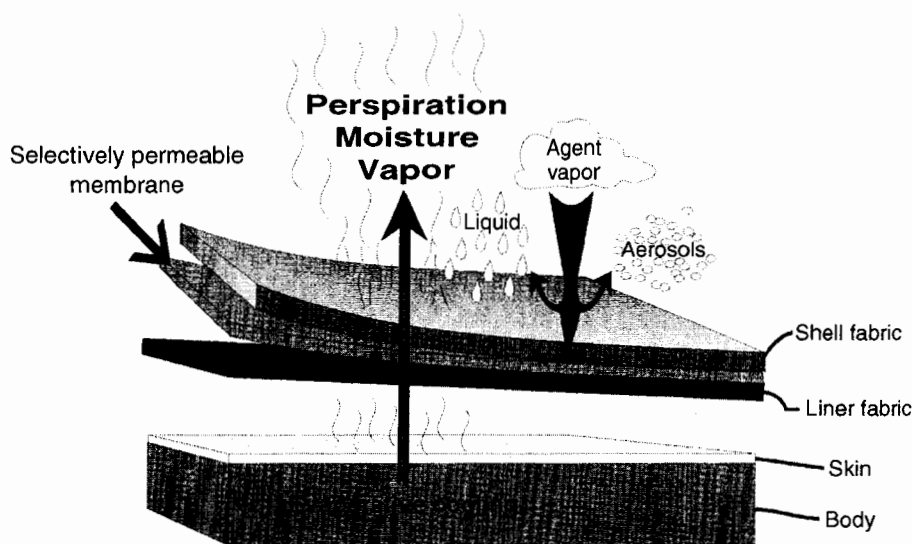


Figure 3. Mechanism of selective vapor transport through selectively permeable membrane fabric.

they can attach onto clothing or gloves in high concentrations and can cause extensive contamination when they leave the surface again as aerosols. Therefore, current efforts are aimed at developing self-decontaminating fabrics that automatically kill any dangerous airborne microorganisms that attach to the protective clothing, in order to avoid endangering nearby unprotected personnel.

Fabric treatments under development to introduce antimicrobial activity covalently bind reactive groups onto fibers and membranes—for example, 4,4-dimethylhydantoin attaches to hydroxymethyl groups at O6 of cellulose to form, after chlorination by hypochlorite, Structure 2 in Chart 1. Other antimicrobial treatments include silver, silver salts, quaternary ammonium groups, and extensively conjugated dyes that act as oxygen photoactivators. Further efforts will be directed toward enhancing

the effectiveness of antibacterial treatments through improvements in chemistry of the fabric itself, modes of attachment, and biocidal mechanisms. Incorporation of biocides into fabrics and on surfaces can help suppress the development of body odor and topical skin and mucosal tissue diseases while affording the wearer a measure of protection against infection resulting from breaks in the skin or by dermal transmission. Commercially available undergarments designed for personal odor control that incorporate the antibiotic Triclosan⁴ (2,4,4'-trichloro-2'-hydroxydiphenyl ether, Chart 1, Structure 1) are good examples.

Unfortunately, several factors inhibit the effectiveness of biocides that act by means of one specific chemical mechanism. First, not all pathogenic species will be dependent on the specific mechanism affected. Second, sensitive species will eventually develop or acquire resistance to the mecha-

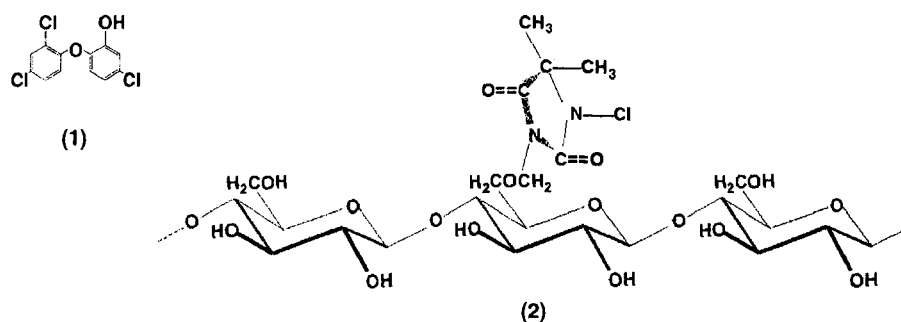


Chart 1. (1) Triclosan. (2) Halamine-treated cellulose.

nism causing toxicity to the contaminating organism (e.g., certain strains of *E. coli* are resistant⁵ to Triclosan). In contrast, solutions prepared from chlorine or iodine remain potentially antibiotic despite generations of use. The toxicity of halogens to microbes presumably results from nonspecific oxidation of functional groups at or near the outer membrane surface of the organism, both on the fabric and in solution. A third liability occurs in select cases wherein the active biocides will eventually be consumed in the disinfection process and protection will be lost; but this is not always the case, as the activity of 2 can be restored by laundry with a hypochlorite bleach. One must note, however, that any treatment applied to garment fabrics must also be tolerated by the wearer, which defines a narrow window of reactivity that an antimicrobial treatment must possess.

N-Halogenated cyclic amides (halamines) are well tolerated by humans and are commonly used as biocides in pools, hot tubs, and drinking water. Similar heterocycles have been grafted to cotton^{6,7} (to produce 2), polyester,⁸ polystyrene, and nylon.⁹ Additionally, with the introduction of appropriate functional groups,¹⁰ such heterocycles can also be incorporated into the manufacturing process for polyester and polyurethane fabric. The modes of attachment to these fabrics and coatings are accomplished with the parent heterocycle, which is converted into the N-chloro derivative (2) by subsequent treatment with dilute solutions of sodium hypochlorite. Functionally, the halamines provide a storage mechanism for chlorine, which is consumed when impinging microbes are killed by oxidation of groups at their surface and when other readily oxidizable species are encountered. However, the Cl can be restored by treatment with dilute bleach during routine laundering, thus eliminating the aforementioned limitations of specificity of action, evolution of microbial resistance, and exhaustion of reactive

capacity. Initial tests on animals¹¹ and small groups of human volunteers showed that the chlorine is well tolerated in these treated materials. Similar antimicrobial activity is expected for treated polystyrene as well as poly(styrene trimethylammonium triiodide) (Triosyn), which can be fused to fabrics or trapped within fibers as a dust. One major drawback of these reactive systems is the apparent photosensitivity of the N-halogen bond. This characteristic will likely limit¹⁰ the use of the N-halamines to undergarments, inner surfaces, and indoor environments.

Because biological agents penetrate fabric and skin much slower than chemical agents, neutralization of surface-held pathogens need not be as immediate as is required for chemical agents. As advanced materials emerge that lengthen penetration times for pathogens, species less reactive than N-halamines—for example, complexed silver (Surfacine), films of elemental silver (Maglon), quaternary cations, and grafts of chitosan—may prove to be useful as incorporated biocides.

In order to provide a high surface area for chemical attachment of new biocides and other reactive compounds onto inner liners of protective fabrics, new microporous membranes are being developed to bind chemically active compounds within the structure of protective fabrics. A new method for forming microporous fibrous membranes is electrospinning. This is a technique of directly spraying submicron-sized fibers onto a collection surface, such as a charged fabric, to produce a microporous membrane. Electrospinning produces submicron fibers from a polymer solution or melt electrically charged to thousands of volts, resulting in a charged jet of fiber from the polymer surface that is fibrillated, stretched in the electric field, and collected on an oppositely charged surface. The resulting mat is a "skin" of randomly oriented fibers with the microscopic appearance of a nonwoven fabric (Figure 4).

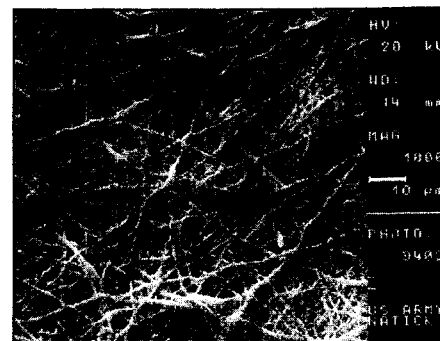


Figure 4. Electrospun polyurethane fibers. Submicron-sized fibers are sprayed directly onto a collection surface, such as a charged fabric, to produce a microporous membrane. Electrospinning produces submicron fibers from a polymer solution or melt electrically charged to thousands of volts, resulting in a charged jet of fiber from the polymer surface that is fibrillated, stretched in the electric field, and collected on an oppositely charged surface. The resulting mat is a "skin" of randomly oriented fibers with the microscopic appearance of a nonwoven fabric.

Fibers electrospun from solutions of thermoplastic polyurethanes (TPUs) have been found to retain good elasticity and strength and also have performed well as matrices for embedded additives that can be used to chemically activate fiber surface reactions.¹² With these TPUs, it is possible to formulate active ingredients into the fibers and use the high surface area of the micro- to nano-sized fibers as well as the high permeability characteristics of the TPUs.

One such active ingredient is *o*-iodosobenzoic acid (1-hydroxy-[4,5]-benziodoxolin-3-one, IBA), which was found to catalytically cleave reactive esters or phosphates.¹³ Hammond et al.¹⁴ demonstrated that IBA derivatives were able to hydrolyze and detoxify nerve agents such as soman, sarin, and tabun. Army collaborators and researchers were able to covalently attach IBA to the oxygen at the C2 position on the secondary ring of β -cyclodextrin to form IBA- β -CD and showed that this compound acted like a pseudo enzyme with the β -cyclodextrin ring supplying a type of active site.^{15,16} This resulted in the catalytic hydrolysis of soman (1,2,2-trimethylpropyl methylphosphonofluoridate) and the simulant dimebu (3,3-dimethylbutyl methylphosphonofluoridate).¹⁶ IBA- β -CD can be incorporated into electrospun polyurethane microfibrils and films and is able to hydrolyze nerve agents in this form.

Recent work has reported on the reactivity of IBA- β -CD and other catalysts

on chemical-agent analogues when the catalysts are incorporated into porous carbon, organic fibers, and film coatings for fabrics.¹² Specialized polyoxometalates (heteropoly acids of the type $H_3PV_2Mo_{10}O_{40}$, POMs), prepared at Emory University, can be adsorbed on porous carbon; this was reported to form selective and recoverable heterogeneous catalysts for the rapid room-temperature oxidation of chemical analogues for mustard (HD).¹⁷ POMs have been incorporated into electrospun polyurethane microfibers and films, and the resulting matrix-bound POMs catalytically decontaminated mustard simulants.¹²

Nanometal oxides recently have been shown to deactivate nerve and blistering chemical agents¹⁸ and can exhibit biocidal activity toward spores of biological warfare analogues.¹⁹ New materials and methods for incorporating protective chemical additives into advanced fabrics are being developed and tested for new-generation protective clothing that will involve not only barrier protection, but also detecting, trapping, and decontaminating toxic particles, liquids, and vapors that contact these advanced fabrics.

Conducting Polymers for Detection

Conducting polymers represent an exciting class of inorganic and organic polymer materials that over the past two decades have been extensively investigated for use in sensor applications.²⁰⁻²² These polymers, which are typically conjugated, can be doped, either chemically or electrochemically, from the insulating state through the semiconducting state to the metallic state.^{23,24} They can also be dedoped through these states, reversibly in many cases. During the doping/de-doping process, many physical and chemical properties are modulated, including the electronic, optical, and magnetic characteristics. In most electronic polymers, doping (de-doping) occurs by partial oxidation (reduction) of the π backbone system of the polymer.²⁵ A number of these polymers are also found to be stable under ambient conditions, for example, polypyrrole, polyaniline, and, more recently, the poly(benzothio-phenes).²⁶ For example, treatment of the polymer with vapors or solutions of oxidizing (reducing) agents can readily accomplish doping (de-doping). Polyaniline represents a unique case in these systems in that doping can also take place simply by protonation of the "emeraldine" oxidation state of the polymer by vapors or solutions of acids. De-doping occurs equally simply from deprotonation by vapors or solutions of bases. Thus, conducting polymers are likely to be highly sensitive to a

variety of properties common to chemical-warfare agents.

A number of groups have investigated the response of the conductivity (resistivity) in these conjugated polymers in the presence of contaminants.²⁰ By monitoring the variation in resistance of a conducting polymer film, electronic sensors for a variety of analytes, including pH, ions, and organic vapors, have been prepared.^{27,28} By combining these sensing elements with more complex detection and chromatographic arrays, it is possible to create what has been termed an "electronic nose," with applications from detecting anaerobic bacterial infections to analyzing wine.²⁹⁻³¹

The application of conducting polymers in fabrics as sensing elements is a new challenge being addressed by the scientific community. Given the known sensitivity of these materials, they make ideal candidates for detection applications. However, a significant challenge involves the specific transduction system to convert the chemical signature into a readout that would provide clear information to the user. Two approaches are currently under investigation related to the use of conducting polymers—nanofibers and optical fibers coated with a conductive polymeric coating.

As resistive molecular wires, nano- and microfibers containing or coated with conducting polymers can be prepared. The electrospinning process mentioned earlier has been well established for insulating polymers and is currently of interest for ballistic protection.^{32,33} However, nanofibrous conductors can also be prepared that can subsequently be woven within a fabric. The first example of this approach involves the blending of the conducting polymer polyaniline with poly(ethylene oxide) (PEO).³⁴ Conducting nanofibers can be prepared by electrospinning a volatile organic solution that contains the base PEO and the emeraldine salt form of the polyaniline. The result is a conducting nanofiber. These fibers have been characterized electronically and shown to respond to gaseous and liquid samples of acids and bases as well as to explosive reducing agents such as hydrazine. An interesting aspect of these high-surface-area materials is the rapid response observed. When compared with thin films of conducting polymers, nanofibers have been found to respond to analytes several times more rapidly. Response times to gaseous analytes such as ammonia were found to be less than 4 s with polyaniline-based materials.³⁵

While resistivity changes produced by the response of a conductive polymer to a stimulus, such as a change in oxidation state in the presence of a chemical contaminant, have been and continue to be effec-

tively detected, changes in the optical properties of a fiber within a fabric can also be detected. Recently, several groups have been examining these changes in order to exploit the advantages of fiber optics for the purpose of sensing chemical-warfare agents. The use of changes in such properties has never previously been investigated for this purpose, although several groups have begun investigating fluorescence responses in conjugated polymers.

The strategy that has been employed by the research groups of El-Sherif at Drexel University and MacDiarmid at the University of Pennsylvania involves chemically etching away the protective cladding from commercial fiber-optic materials and replacing it with a chemically responsive conducting polymer such as polyaniline or polypyrrole.^{36,37} The deposition of the polymer was achieved using an *in situ* polymerization process that allowed for control of the film thickness and level of doping. The submicron-thick films of polyaniline on the optical fibers change upon reaction with acidic, basic, oxidative, and reductive vapors. The chemical interaction results in (1) a change in the refractive index and (2) a change in the optical-absorption spectrum of the polymer film. Both of these changes modify the intensity of the light transmitted through the optical fiber as a result of changes in the refractive index and interactions with the evanescent field, respectively. Changes in optical-fiber throughput from 5–95%, depending upon the specific analyte, were observed. By variation of the conducting polymer and additional investigation with the addition of catalysts such as bis-bipyridyl copper nitrate complexes,³⁸ this approach was found to respond positively to nerve agent simulants such as dimethyl methylphosphonate (DMMP) and diisopropyl fluorophosphate (DFP).³⁹

An extension of conducting nanofibers involves the creation of molecular tubes, which are interesting for sensor applications because of their high surface-to-volume ratios and potentially enhanced sensitivity. While carbon nanotubes dominate this nanotechnology area, conjugated conducting polymers have attracted great interest for potential application in molecular devices. Several preparation methods of micro- or nanotubes of conducting polymers reported in the literature include template synthesis and template-free methods.³⁹⁻⁴⁴ Recently, a novel fiber-templating approach to prepare conducting polymer or metallic tubes was described that allows for the flexible creation of conducting polymer and solid-state tubes.⁴⁴ This new approach involves nanometer-diameter polymer fibers prepared by electrospinning as templates on which subsequent materials

can be grown. Thermal treatment of the composite coaxial fiber removes the core polymer, leaving behind a nanotube of the outer metal or conducting polymer material (Figure 5).

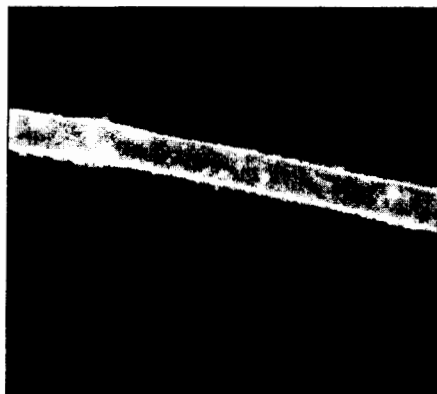


Figure 5. Gold tube with a wall diameter of 50 nm, after thermal removal of polystyrene nanofiber core. This represents a next-generation nanowire material that could be incorporated into future smart fabric designs.

Conclusions

Selectively permeable materials have been developed as garment fabrics that provide effective protection against chemical and biological agents while maintaining good moisture-transport properties at a lower weight with less bulk than previous designs. Protective, reactive, and responsive fabric technologies will continue to advance with the development of new membranes capable of reactive self-decontamination as well as detection of toxic contaminants. Incorporation of biocides and chemically deactivating compounds into these advanced fabrics will provide self-detoxifying clothing systems in the future. Detection by means of new conductive fibers and cladding materials will give the wearer an early warning of environmental threats. Such features will be available for future-generation protective clothing that will keep soldiers and emergency responders safe with enhanced pro-

tection against chemical and biological agents in toxic or contaminated environments.

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